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U. S. DEPARTMENT OF AGRICULTURE
Agricultural Research Service

NEW TEXTILE PRODUCTS FROM COTTON

Partially
Acetylated
COTTON

By

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FOREWORD

The information summarized herein on the nature, properties, uses, availability, and estimated costs of PA (partially acetylated) cotton, a new textile fiber, is presented to help answer many questions concerning this material.

These data will be of interest to those desiring general knowledge about PA cotton. Additional and more detailed information will be made available to those requesting it. The statements made herein are expressions of our opinion, based on research and experience in the field. Those using this information do so with the understanding that we cannot accept liability for performance in any given application.

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1/ The Southern Utilization Research Branch (SURB) comprises the Southern Regional Research Laboratory (SRRL) at New Orleans and its six associated field stations in Florida, North Carolina, Louisiana, and Texas. SURB is one of the branches of the USDA Agricultural Research Service.

PA (PARTIALLY ACETYLATED) COTTON

Page

Fibrous Cellulose derivatives from cotton 4

Chemical nature of PA Cotton 8

Properties 10

Present uses 18

Potential uses 18

Availability 20

Estimated costs 20

Additional textile products from cotton 21

Cotton as a raw material for the chemical industry 21

Bibliography 23

FIBROUS CELLULOSE DERIVATIVES FROM COTTON

In producing new textile fibers, industry has given great attention to methods characterized by the two operations of (1) conducting chemical reactions in solution to obtain the desired chemical composition followed by (2) extrusion to create the fibrous form. Relatively little effort has been devoted to the converse esequence of operations, i. e., (1) creating the fibrous form followed by (2) effecting chemical reactions on or with the fibers to arrive at the desired chemical composition.

While the second method has received relatively little attention, it offers many attractive possibilities when applied to cotton. Not only is cotton a fiber of great value in its own right, but it also is inexpensive and unusually versatile in its ability to undergo changes when treated with chemicals. New fibers made in this manner are synthetic products having new chemical, physical, and textile properties. Partially acetylated cotton, called

PA COTTON

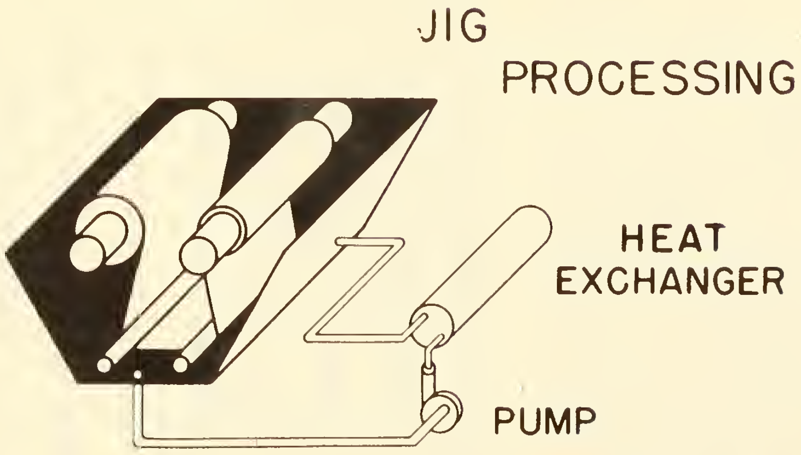
is the first practical fibrous chemically modified cotton investigated intensively at the USDA Southern Regional Research Laboratory. It has received considerable attention by the cotton industry and others during recent years because of its outstanding resistance to deterioration by micro-organisms and heat, resulting in unusually long service life in tests of hot-head press covers in commercial launderies and in household ironing board covers, and in tests of sandbags exposed on damp ground, bags for holding the chemicals in home water-softening systems, and nets and twines for fishing gear in river and ocean waters.

The process for producing acetylated cotton uses acetic anhydride and a catalyst to convert the accessible cellulose of cotton to an acetate. The modified cotton remains nontoxic and is not changed in general appearance. Unlike some processes used to improve the rot resistance of cotton, partial acetylation leaves no stickiness, odor, or color. It is believed to be the most effective method developed to protect cotton from damage by heat and rot-causing micro-organisms.

PA cotton retains the fibrous structure of the original cotton in contrast with other forms of cellulose acetate in the formation of which the cotton fibers are dissolved and the solution then extruded or shaped into rayon, plastics, or films.

Practical processes for the production of PA cotton from raw stock, yarn, or fabric have been developed at the USDA Southern Regional Research Laboratory (SRRL) and are the bases of processes adopted by industry in this country. These processes include the batch acetylation of raw stock, yarn, and fabric, and the continuous acetylation of yarn and fabric. Typical applications of the batch and continuous processes for fabric are illustrated in Figures 1 and 2: Equipment and handling techniques are largely conventional to either the chemical or textile industries. Information as to the adaptation of these processes to specific products can be obtained from the Southern Utilization Research Branch.

SRRL-based processes are the first to be developed in the United States for commercial use. Applications based on the heat and rot resistance of PA cotton were developed first by SRRL. However, processes of partial acetylation did not originate at SRRL. British scientists in 1901



ACTIVATION	REACTION	WASH
Glacial Acetic Acid	85% Glacial Acetic Acid	Water
1 hr., room temp.	15% Acetic Anhydride	
	0.15% Perchloric Acid	
	1 hr., 68° F.	

Figure 1. Diagram - jig acetylations.

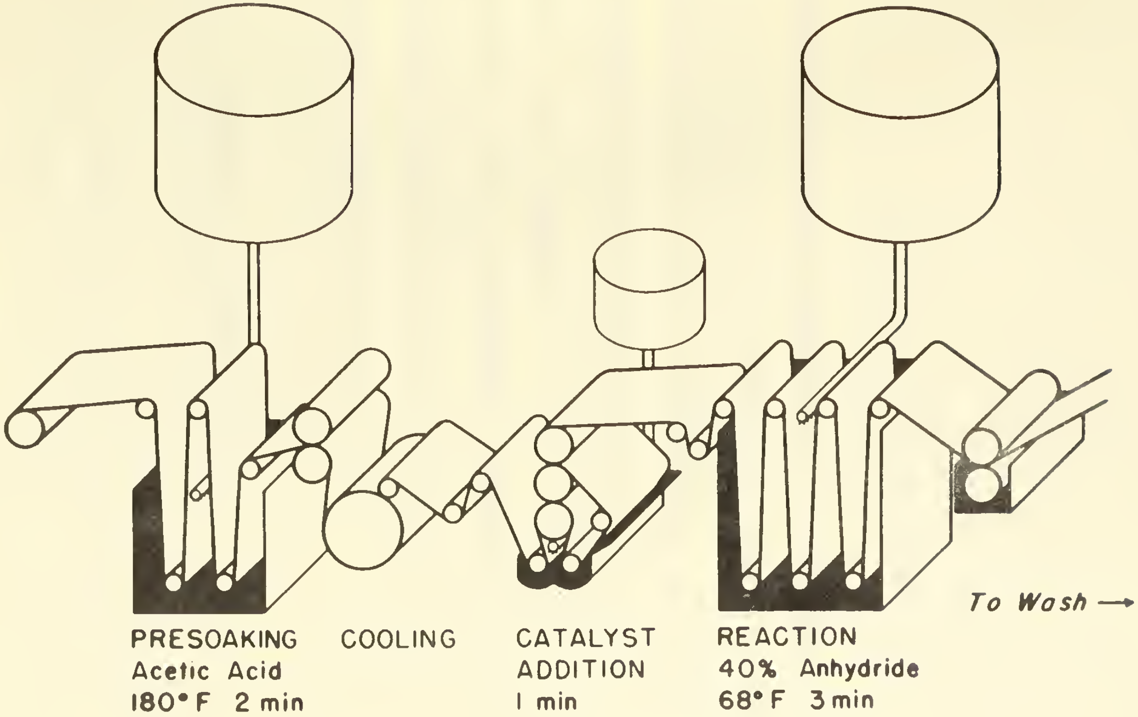


Figure 2. Diagram - continuous acetylation range.

reported the earliest preparation of a fibrous partially acetylated cotton yarn (14) 2/ and an acid-catalyzed process for producing it was patented in 1933 (21). Another British product, described in earlier patent specifications as being produced by a zinc-chloride-catalyzed process, is called Cotopa 3/ (or Crestol, if applied to mercerized cotton). These products are understood to be in commercial production in England for special uses (19, 20).

CHEMICAL NATURE OF PA COTTON

With the proper catalysts and conditions, all the hydroxyls of the cellulose that composes cotton can be esterified through reaction with acetic anahydride, that is, the hydrogens of each hydroxyl on the anhydroglucose units can be replaced by acetyl groups. In practical applications substitution at about one-third, or even less, of all the sites is adequate, the main requirement being acetylation of the more readily accessible hydroxyls in the amorphous regions and crystallite surfaces. The product is believed to be a structure retaining most of the original crystalline cellulose, with all, or almost all, of the interspersed accessible cellulose converted to the triacetate, averaging the direct substitution of one acetyl per glucose unit (DS=1) or less through the entire mass. This composition will be used as a basis for the discussion of fibrous PA cotton. Fibers of cotton acetylated to an average of DS=1 for the entire fiber by the methods described almost invariably appear uniform when dyed and examined with the microscope.

2/ Figures in parentheses refer to Bibliography, p.23-24.

3/ It is not the policy of the Department to recommend products of specific companies over those of any others engaged in the same business. Their names are furnished merely for convenience and information.

There is nothing to suggest a distribution of acetyls resembling a protective layer or sheath around each fiber. A full discussion of the nature of these products has already been published (6).

The degrees of substitution of acetate rayons and partially acetylated cottons may overlap, but their properties are greatly different. Even among acetylated cotton products, which have retained their fibrous nature throughout processing, there are great differences between products obtained by preparing partial acetates approached from the cotton side, as opposed to those prepared by deacetylation, or saponification, from a higher value, as illustrated in Table I. It is seen, for example, that with two products of the same average composition, approximately one-third substituted, the one prepared by acetylation up to that value behaves almost like the nearly completely substituted product in dyeing, swelling capacity, and rot resistance; the other, prepared from a higher substituted product by deacetylation, dyes, retains water, and rots very much like ordinary cotton.

TABLE I

EFFECT OF ACETYLATION AND DEACETYLATION ON PROPERTIES OF COTTON

Sample acetyl	DS	Dyeing**	Swelling Capacity***	Strength after burial 0 days	Strength after burial 14 days	Strength after burial 6 mos.
%			%	%	%	%
0	0	blue	38	100	0	0
23	1.1	yellow	23	100	100	100
41	2.6	yellow	20	106	104	98
23*	1.1	blue-green	32	100	0	0

* Deacetylated from DS=2.2, 37% acetyl.
** A direct blue and an acetate yellow in same bath.
*** Water held on centrifuging at 5820 times gravity.

PROPERTIES

PA cotton is odorless, nontoxic, and unchanged in color. Its appearance, breaking strength, and texture differ only slightly from those of untreated cotton. Higher average degrees of acetylation (above DS=1) do, however, produce some stiffness. A number of other properties are decidedly changed.

The improved or changed properties of PA cotton indicate that it not only is better suited for a number of the conventional uses of cotton, but that it is also suitable for markets for which ordinary cotton is poorly suited. The properties of PA cotton and untreated cotton are compared in Table II. Improved or changed properties of PA cotton are given below.

Increased Heat Endurance

Of great commercial interest is the heat endurance of fibrous partially acetylated cotton materials. Figure 3 gives typical heat-endurance figures. In this graph is shown the strength of acetylated yarn for oven exposures at two different temperatures plotted against time on a logarithmic scale. At 250° C (482° F) cotton is reduced to 2/3 strength in 3 minutes, while comparable yarn made up from acetylated raw stock is down to 2/3 strength only after 25 minutes, about 8 times as long. Similar comparison at the 1/2-strength criterion gives acetylated cotton a 6 times advantage. Likewise, at 160° C. (320° F.) acetylated cotton yarn lasts 12 to 15 times as long as untreated cotton. It is not surprising that, in laundry-press-pad covers, acetylated sheetings give 4 to 5 times the service life of cotton canvas.

Mildew and Rot Resistance

PA cotton is highly resistant to attack by micro-organisms. The following service tests have established the extent of its resistance:

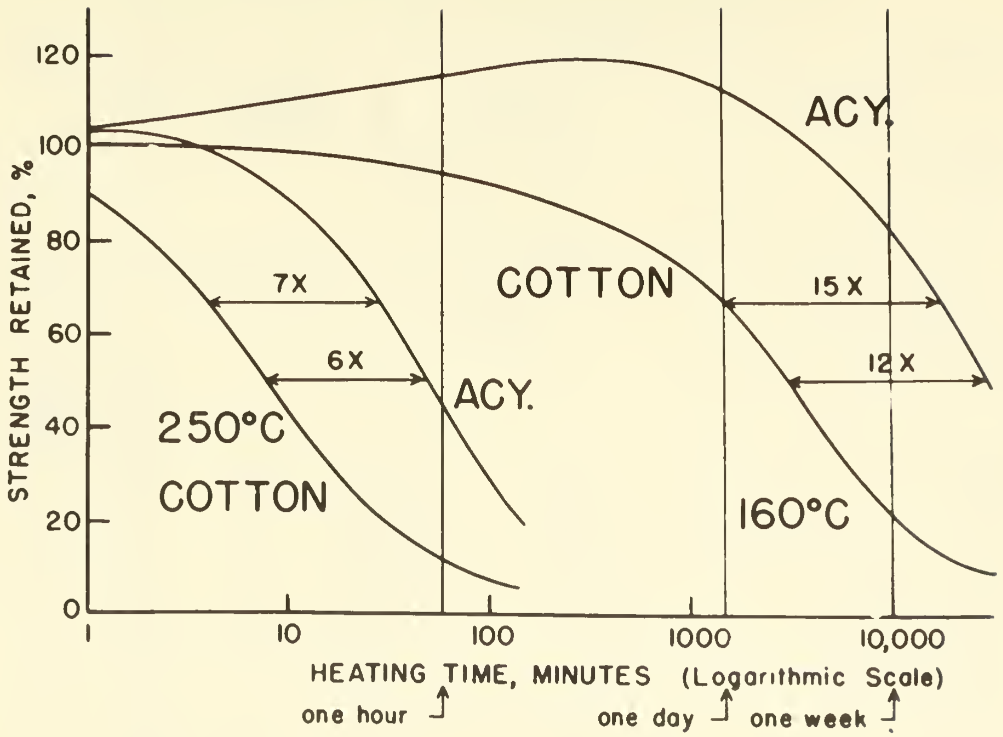


Figure 3. Heat endurance of partially acetylated cotton.

TABLE II

COMPARATIVE PROPERTIES OF PA COTTON AND UNTREATED COTTON

Property, Units	Untreated cotton	PA cotton	PA Cotton finished as noted
Fiber properties (9)			
Tenacity, 70°F., about 1% r.h., g./den	2.4 - 2.6	2.5 - 2.6	-
Tenacity, std. cond. g./den	3.2 - 3.5	2.6 - 2.8	-
Tenacity, wet, g./den	4.0 - 4.3	2.6 - 2.8	-
Moisture regain, std. cond., %	6 - 7	3 - 3.5	-
Elongation at break, std. cond., %	7 - 10	6 - 9	-
Elongation at break, wet, %	12 - 14	-	-
Specific gravity	1.55	1.43	-
Fabric properties 1/			
Breaking strength, std. cond., lb.	50	45 - 55	45 - 55
Weather endurance, strength retained after 1 year exposure, % of original	35	75 2/	75 2/
Melting point	does not melt	does not melt	does not melt
Hot iron sticking temperature	does not stick	250°C.	250°C.
Heat endurance, strength retained after 2 weeks in 130°C. oven, % of original	48	94	94
Scorch appearance,/under 250°C. iron for 5 min.	black	ecru	ecru 3/
Flex-abrasion endurance, Stoll, cycles	699	120	999 4/
Flat abrasion endurance, Stoll, cycles	180	196	165 - 185 4/
Trapezoid tear strength, lbs.	13.3	8	12.0 4/
Elmendorf tear strength, lbs.	10.2	4.3	6.4 4/
Rot resistance, strength retained after 2 weeks in active/burial beds, % of original	0	100	100
Rot resistance, strength retained after 26 weeks in active/burial beds, % of original	0	95 - 100	95 - 100

1/ For a scoured cotton sheeting, 4 oz./sq.yd. before treatment, 4.5 oz./sq.yd. after treatment, 20% acetyl content (typical values).

2/ Vat dyed with selected colors before acetylation (I).

3/ Change in shade undetectable on fabric dyed with 5% (total) Ponsol Browns ARD and AGD before acetylation. See footnote 3/, page 8.

4/ After treatment with selected lubricating agents durable to washing and heating.

(1) Soil burial: Typical results obtained on burial of rot-proofed cotton fabrics in highly active soil are shown in Figure 4. Under the conditions used at the Southern Regional Research Laboratory, untreated cotton loses practically all its strength in 1 week; fabrics treated with copper-containing additivies are reduced to 30% of original strength in 8 to 12 weeks; and properly acetylated goods retain 80 to 100% strength for longer than 50 weeks. Actually, a small gain in strength is often observed in acetylated cotton during the early stages of these burial tests; this effect is paralleled in heat tests as well (Figure 3).

(2) Exposure in ocean water: In continuous exposure up to 8 months in ocean water, PA cotton twines of the type suitable for making fishing nets lasted 2 or 3 times as long as a tar control and at least 8 or 10 times as long as untreated cotton twines (Table III).

TABLE III

STRENGTH RETAINED BY ACETYLATED AND UNACETYLATED FISH NET TWINES IN OCEAN WATER*

Nature of Twine	Strength Retained After Exposure				
	1 mo.	2 mo.	4 mo.	6 mo.	8 mo.
	%	%	%	%	%
Untreated	15	0	0	0	0
Tarred control	94	85	42	1	0
Acetylated	102	109	107	102	84

*Tested by E. W. Roelofs, courtesy of Institute of Fisheries Research, University of North Carolina, Morehead City, N. C.

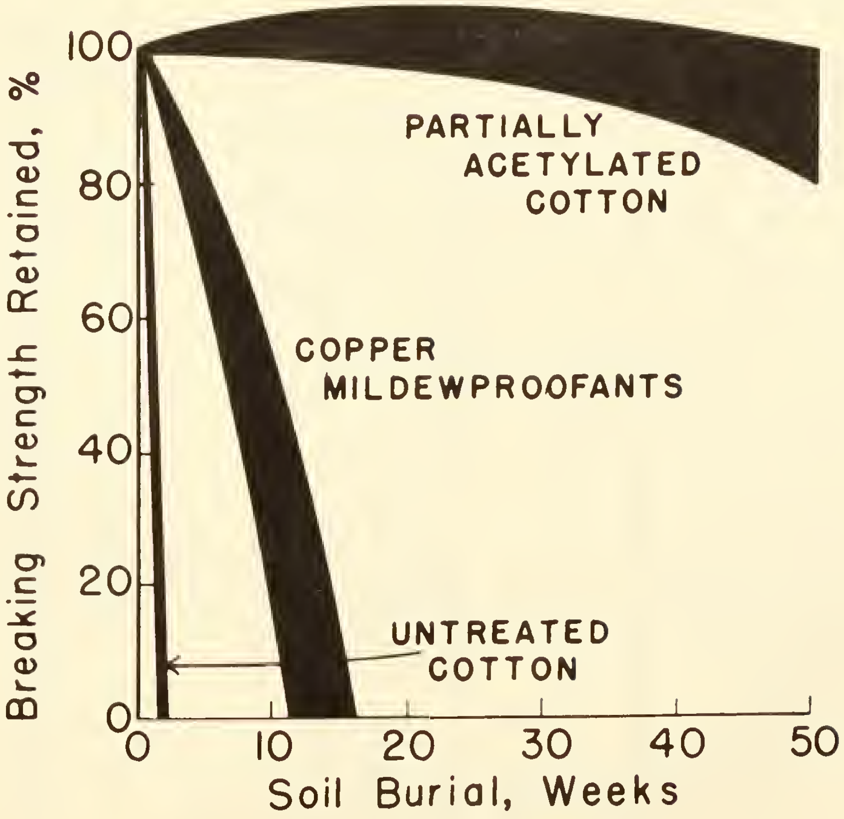


Figure 4. Typical results of SRRL burial tests on treated cottons.

(3) Sandbag tests: Sandbags made from acetylated cotton cloth of 29% to 30% acetyl using acetylated thread, in contact with the ground at the bottom of a pile, remained intact after 2 years and retained 35% to 40% of their breaking strengths. The surface fabric of the bags at the tops of stacks retained 35% to 100% strength, compared to complete deterioration by untreated bags. In comparative cases, thread of 23% to 25% acetyl content after the 2 years' exposure in seams in the bags retained 63% to 86% of its strength, while thread treated with 14% of copper naphthenate had completely disappeared, or was so weak that it could not be recovered for testing.

Weather Resistance

PA cotton behaves substantially the same as untreated cotton on exposure to weather. A recent accomplishment of much promise combines resistance to sunlight with mildew- and rot-resistance of PA cotton. The method consists of predyeing cotton with certain light-stable, nonsensitizing vat-dyes, followed by partial acetylation. Weather exposure tests indicate that the combination of vat-dyeing with certain colors and acetylating may double or treble the outdoor service life compared to cotton vat-dyed only.

Changed Dyeing Properties

PA cotton resists coloring with many direct dyes but can be dyed in a complete range of colors by acetate dyes. This characteristic is the basis of using PA cotton to obtain dye-resist and cross-dyeing effects. In some instances it is preferable to vat-dye cotton prior to acetylation (1).

Lower Moisture Regain

The moisture regain of PA cotton is significantly less than that of ordinary cotton. For products near DS=1 the regain is about 3% to 3.5% in contrast to about 7% for untreated cotton under standard conditions. Water retention on centrifugation is also reduced to about one-half that of untreated cotton.

Resistance to Chemical Attack

Acid and alkali resistance: PA cotton resists acid attack better than untreated cotton. In one test, PA cotton yarn lost only about one-third of its strength in 8 hours in 20% hydrochloric acid, whereas untreated cotton lost two-thirds. Under strongly alkaline conditions, progressive losses in strength up to about 24% are observed as deacetylation takes place, and, of course, PA cotton loses its distinctive properties. This takes place in 1 day in 2% sodium hydroxide (pH above 13), but requires more than 1 week in 3.5% ammonium hydroxide (pH about 12).

Resistance to corrosive atmospheres: Exposure of partially acetylated cotton to nitrogen dioxide and air mixtures and to hydrogen chloride and air mixtures showed that this chemically modified cotton is resistant to the action of these gases as compared to ordinary bleached cotton. For example, in a 3% mixture of hydrogen chloride in air, ordinary bleached cotton loses about two-thirds of its strength in 6 hours, while PA cotton loses only about 6%. Similarly, in a 2% mixture of nitrogen dioxide in air, the losses are approximately 70% and 30%, respectively (10).

Electrical Characteristics

In common with other substances having a similar chemical composition, such as acetate rayon, acetylated paper, and cellulose acetate plastics, direct current insulation resistance of PA cotton is many thousand times that of ordinary cotton, depending on the degree of substitution, extent of washing, and the humidity of the ambient atmosphere. Alternating current characteristics, such as power factor and permittivity, are also considerably improved (18).

Dermatological Effects

Garments were made of PA cotton and were worn for extended periods in contact with the skin. Wearers have reported no irritating or other harmful effects.

Mechanical Processing Characteristics

Partially acetylated cotton stock in thirty-pound and smaller lots has been satisfactorily spun into yarn through the use of selected anti-static agents which have no adverse effect on the heat endurance of PA cotton. Promising results were also obtained using blends of partially acetylated (80%-90%) and untreated cotton (20%-10%). The results of these experiments with small lots indicate that most of the major mechanical processing problems encountered with PA cotton fiber can be satisfactorily solved by the use of selected additive finishes, or by blending with a moderate percentage of untreated fiber (11).

Other Properties

Resistance to insects: There has been no evidence of attack by insects on PA cotton during years of storage.

Tear resistance: Tear strength and flex abrasion of cotton fabrics are reduced by acetylation, but these losses can be largely overcome by fabric design, or by lubricating or softening the modified cotton with selected softening agents. The durability of these softeners in service applications is currently being investigated; a number are available which are permanent to washing and heating.

Soil resistance and launderability: Soil resistance and ease of removal of soil from cotton fabrics are improved by partial acetylation. PA cotton may be laundered without difficulty if neutral detergents are used. With common soaps, the effects of hydrolysis, especially on heat resistance, become noticeable after two or three wash cycles.

Absorbency and drying: Water as measured by washing and rate of drying of PA cotton are similar to those characteristics of untreated cotton.

Flammability and effects of common organic solvents on PA cotton: These are generally the same as those for untreated cotton.

PRESENT USES

PA cotton, processed in accordance with the methods developed in the USDA Southern Utilization Research Branch, is produced and marketed in the United States under commercial trade names for household ironing board covers. It is being evaluated for various other uses by a number of industrial organizations.

Other acetylated cottons, produced by another process, are being sold by an English firm.

POTENTIAL USES

Some of the major potential uses for partially acetylated materials are listed in Table IV, along with recent cotton consumption figures in bales. It is considered probable that, for a substantial part of these uses, cotton could be partially acetylated to give products which would be in a better competitive position than cotton itself on a basis of cost per unit of service life. While it would not be expected that all

TABLE IV

U. S. COTTON CONSUMPTION IN END USE MARKETS IN WHICH PA COTTON SHOWS ADVANTAGES

End uses	Cotton market* (478-lb. bales)
Auto tops, convertible	2,720
Awnings	43,720
Barricade and sandbags	-
Coated truck fabric	6,250
Conveyor belts	17,230
Diapers, collection service	-
Filter fabrics	20,570
Flags	4,200
Flat-work-ironer aprons	10,250
Flat-work-ironer cover cloths	26,870
Flat-work-ironer feed ribbons	420
Flat-work-ironer padding	10,600
Home-ironer covers	-
Hot mill gloves	6,730
Industrial thread	142,600
Insulation, electrical	130,210
Insulation, thermal	150
Laminate fabrics	-
Net and seine twine	58,680
Paper-dryer felts	-
Press-bed covers	10,930
Press-bed flannels	24,240
Press-bed padding	12,020
Shoe linings, leather	62,300
Shoe fabrics, rubber	40,440
Shower curtains	890
Splash and wading pools	-
Tarpaulins	93,640
Tents	35,290
Umbrellas, beach and garden	1,480
Water-softener bags	-
Zipper tape	8,080
Total	770,510

*National Cotton Council of America revised estimates materials in these categories would be replaced by partially acetylated cotton, these figures nevertheless suggest great possibilities.

AVAILABILITY

PA cotton is available in the United States for commercial use from the following companies[4]:

Proctor Electric Company Philadelphia, Pennsylvania	Wm. E. Hooper & Sons Company Baltimore, Maryland

ESTIMATED COSTS

An estimate of the cost of PA cotton yarn produced by a continuous partial acetylation process has been made primarily on the basis of currently available information obtained in pilot-scale operations (4). Preliminary cost estimates of producing PA fabric by the continuous process and of producing PA stock, yarn, and fabric by batch processes, indicate that the processing costs for these different forms and types of processes will not differ greatly.

The total processing cost for partially acetylating desized cotton sheeting (60-inches wide, 8-ounces per running yard) by the continuous process is estimated to be about 21 cents per yard (42 cents per pound) of material processed to an approximate composition of 15 percent acetyl content. This estimate of total cost is based on processing about 1-1/4 million yards of fabric annually, operating 16 hours per day, 250 days per year with a working capital of about 30% of the fixed capital investment of approximately $240,000, and amortization of the major portion of the special equipment in 15 years. Estimates show that at an annual production rate of the order of 15 million pounds or greater the recovery of acetic acid from wash water (amounting to about one

[4] See footnote 3, page 8.

pound of acid per pound of cotton processed) should become profitable, thereby effecting a considerable reduction in costs. On the other hand, as production goes down, costs rise, and processing costs might be as high as 40 cents per yard, or even higher, for this material depending upon the degree of intermittent operation. Higher costs are, therefore, to be expected in the early stages of market development and in small-scale or intermittent operation.

ADDITIONAL TEXTILE PRODUCTS FROM COTTON

Listed in Table V are other chemically modified cottons developed or investigated by the Southern Regional Research Laboratory. Additional information about these will be supplied upon request.

COTTON AS A RAW MATERIAL FOR THE CHEMICAL INDUSTRY

Cotton, first grown in North America in the Colony of Virginia in 1607, is now grown on more than a million farms in the southern and western parts of the United States. The average size of the crop is about 13 million bales of cotton lint (each weighing about 500 pounds). In the past 30 years, the largest crop was 18,945,028 bales (about 9 billion pounds) in 1937; the smallest 8,639,595 in 1946.

In the colony of Virginia cotton was quoted at 8 pence a pound. The season average farm price for the basic grade and staple length of American Upland cotton for the crop year 1955 was 32.44 cents a pound. Since the end of World War II, the highest average annual price was 42.58 cents a pound in 1950; the lowest 31.83 cents a pound in 1949.

Improved production methods are bringing increased yields and the prospect of further reductions in the cost of production. These factors are making cotton more and more an attractive raw material for the chemical industry.

TABLE V

OTHER NEW PRODUCTS FROM COTTON THROUGH CHEMISTRY*

Type of Product	Reagents or Treatment	Principle Advantages
Elastic bandage and fabrics	Slack mercerization	Improved elongation and elastic properties, kinkiness, cling
Tobacco shade cloth	Lead Chromate	Improved resistance to deterioration by sunlight
THPC cotton	Phosphorous compound	Flame resistant; also rot- and crease-resistant
Decrystallized cotton	Ethylamine	Greater elongation and reactivity
Aminized cotton (AM cotton)	Aminoethyl sulfuric acid	Enhanced dye receptivity and chemical reactivity
Carboxymethyl cotton (CM cotton)	Chloroacetic acid	Enhanced reactivity and water solubility
Cyanoethylated cotton (CN cotton)	Acrylonitrile	Improved receptivity to dyes and improved resistance to heat and rot
Propiolactone cotton (PL cotton)	Propiolactone	Wool-like feel
BAP cotton	Bromine-containing organo phosphorous compound	Flame resistant

* The first three products are commercially available. AM cotton, CN cotton, and BAP cotton have been made in large experimental quantities.

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